United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

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Title: LOD and LOQ for Chromatographic Methods		
Revision: 3	Replaces: 07/01/04	Effective: 10/01/06

1. Purpose:

To provide guidance for estimating and establishing the limit of detection (LOD) and limit of quantitation (LOQ) for analytes reported to the USDA/AMS Pesticide Data Program (PDP). PDP LODs are method-driven and are experimentally determined by fortifying and analyzing duplicate matrix samples.

2. Scope:

This standard operating procedure (SOP) shall be followed by all analytical laboratories conducting residue studies for PDP, including support laboratories conducting stability or other types of studies that may impact the program.

3. Outline of Procedure:

5.1 Method Noise
5.2 Establishment of LOD
5.3 Establishment of LOQ

4. References:

- USDA/AMS PDP QA/Technical Meeting, May 18-20, 2004, Fairfax, VA
- USDA/AMS PDP QA/Technical Meeting, May 7-9, 2003, Manassas, VA
- PDP QC-10 drafts, May 1995-January 1996
- Quality Assurance Committee conversations, March 1995-January 1996
- Quality Assurance Officer's Meeting, February 21-23, 1995
- FDACS QA/QC Guideline Document, Section 14
- Letter, Martha Lamont to PDP participants, May 5, 1992
- GLP Meeting with USDA/AMS GLP Committee and Robert Epstein, 4/29/92
- GLP Meeting with EPA/OPP, EPA/OCM, USDA/AMS, and USDA/AMS GLP Committee, 4/28/92

- Chemist Qualification document from Robert Epstein and summarized by Terry Jackson with State participant comments, 4/23/92
- Quality Assurance Principles for Analytical Laboratories, Garfield, F., AOAC, 1991
- Validation of Methods Used in the Florida Department of Agriculture and Consumer Services' Chemical Residue Laboratory, Parker, G.A., JAOAC, 74, No. 5, pp. 868-871, 1991
- Quality Assurance of Chemical Measurements, Taylor, J.T., Lewis Publishers, 1989
- Evaluation of Analytical Methods Used for Regulation of Foods and Drugs, Horwitz, W., Analytical Chemistry, Vol. 54, No. 1, pp. 67A-76A., 1982

5. Specific Procedures:

5.1 Method Noise

5.1.a Method noise is the combination of instrument noise and the matrix noise contributions.

5.1.b Method noise determination must be completed for all required PDP analytes.

5.1.c Method noise will be determined utilizing instruments and operating conditions which are routinely used for the analysis of samples. Noise for the LOD and LOQ calculations will be determined by examining chromatograms of the blank commodity in the chromatographic time segment (CTS) of the pesticides of interest.

5.2 Establishment of LOD

5.2.a LOD may be estimated by multiplying the response of method noise level by approximately three and then converting the total response into concentration, i.e., ppm, ppb, or ppt.

For example: 1) take two equal portions from the same matrix blank extract; 2) spike one aliquot with a known amount of the analyte of interest; 3) inject both aliquots under the same conditions; 4) magnify the baseline of the unfortified blank at the analyte retention time window of interest to obtain the instrument response for the tallest (height) or the broadest (area) noise; and 5) convert the response into concentration (ppm, ppb, or ppt) from the known concentration of the spiked extract.

5.2.b LODs may be established at a level greater than three times noise.

5.2.c The reported LOD shall be the highest value obtained using the primary identification technique and the confirming technique.

Examples:

1a) Identification: GC NPD with a particular column.
2b) Confirmation: GC MSD with an appropriate alternative column.

2a) Identification: GC FPD with a particular column.
2b) Confirmation: GC ELCD with an appropriate alternative column.

3a) Identification: GC ELCD with a particular column.
3b) Confirmation: GC ELCD with an appropriate alternative column.

5.2.d LOD is method dependent and shall be experimentally verified in matrix as detailed in SOP PDP-QC-07, Demonstration of Method Performance.

5.3 Establishment of LOQ

5.3.a LOQ will be calculated/determined for each analyte in each commodity tested following the establishment of LOD.

5.3.b For all detection systems other than mass spectrometry, LOQ will be established by multiplying the response of method noise level by at least ten and then converting the total response into concentration (i.e., ppm, ppb, or ppt), or by multiplying the LOD by no less than ten/thirds (10/3) if the LOD is established above three times method noise. Confirmation criteria shall be met.

5.3.c For mass spectrometric systems, ions to be used for qualitative analysis/confirmation shall be at least three times signal to method noise. Ions to be used for quantitation shall be at least ten times signal to method noise. Confirmation criteria shall be met.

5.3.d The reported LOQ shall be the highest value obtained using the primary identification technique and the confirmation technique.

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Revision 3 February 2006 Monitoring Programs Office

- Changed title of SOP from “Estimation of LOD and LOQ for Chromatographic Methods” to “LOD and LOQ for Chromatographic Methods”
- Updated format to conform with other SOPS
- Renamed subsections 5.1, 5.2, and 5.3
- Added description of method noise to subsection 5.1
- Deleted subsection 5.1.c and 5.1.d
- Deleted subsection 5.2, Guidelines for Estimation of Method Noise in Chromatographic Systems and renumbered remaining subsections
- Added subsection 5.2.d
- Changed “will” to “shall” in subsections 5.2.c and 5.3.b
- Moved requirements of subsection 5.4.c into 5.3.a

Revision 2

- Changed title of SOP from “Determination of LOD and LOQ for Chromatographic Methods” to “Estimation of LOD and LOQ for Chromatographic Methods”
- Moved procedures for determination/verification of LOD to Subsection 7.4 in SOP PDP-QC-07, “Demonstration of Method Performance”
- Added estimation of LOQ for mass spectrometry systems to Subsection 5.4.c